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6,6,8a-Trimethyl-3a,6,7,8a-tetrahydrobenzo[*b*]furo[3,2-*d*]furan-2,4(3*H*,5*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.068
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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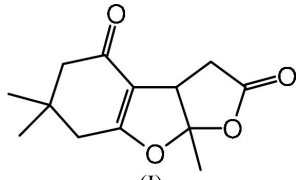
The structure of the title compound, $C_{13}H_{16}O_4$, comprises a non-planar chiral molecule where the cyclohexene double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å).

Comment

The title compound, (I), a perhydrofurobenzofuran, exhibits hypoglycemic properties. A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) for related structures reveals that there are 38 compounds containing a six-membered carbocyclic ring with two linked five-membered furo rings, as in (I). However, in all 38 molecules the C_6 ring is benzene; none are cyclohexane, -ene or -yne variants. The structure of (I) comprises a non-planar chiral molecule where the C5═C10 double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å). The two torsion angles that highlight the non-planarity of the molecule are O1—C1—C4—C3 [−127.2 (1)°] and O2—C1—C4—C5 [104.0 (1)°].



(I)

Experimental

The title compound was prepared according to the literature procedure of Nagarajan *et al.* (1988). Crystals were grown from ethanol.

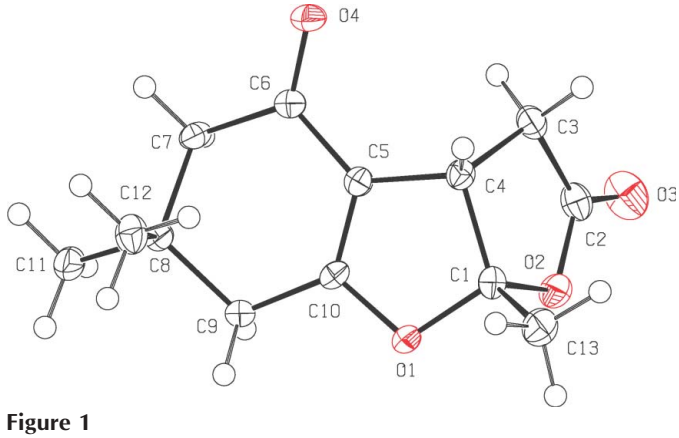


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

Crystal data

$C_{13}H_{16}O_4$	Mo $K\alpha$ radiation
M_r = 236.26	Cell parameters from 1560 reflections
Orthorhombic, $P2_12_12_1$	θ = 2.9–27.5°
a = 9.4853 (3) Å	μ = 0.10 mm^{-1}
b = 10.2904 (2) Å	T = 120 (2) K
c = 12.2872 (4) Å	Prism, colourless
V = 1199.32 (6) Å^3	0.50 × 0.40 × 0.40 mm
Z = 4	
D_x = 1.309 Mg m^{-3}	

Data collection

Nonius KappaCCD diffractometer	2235 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.021
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	θ_{max} = 26.0°
T_{min} = 0.953, T_{max} = 0.962	$h = -11 \rightarrow 10$
8608 measured reflections	$k = -12 \rightarrow 12$
2343 independent reflections	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.2043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.068	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
2343 reflections	Extinction correction: *SHELXL97*
158 parameters	Extinction coefficient: 0.045 (6)
H-atom parameters constrained	

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.98 (CH_3), 0.99 (CH_2) and 1.00 Å (CH). The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom. In the absence of significant anomalous scattering effects, the 763 Friedel pairs were merged.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. J. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Nagarajan, K., Talwalker, P. K., Goud, A. N., Shah, R. K., Shenoy, S. J. & Desai, N. D. (1988). *Indian J. Chem. Sect. B*, **27**, 1113–1123.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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Crystal data

$C_{13}H_{16}O_4$
$M_r = 236.26$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.4853$ (3) Å
$b = 10.2904$ (2) Å
$c = 12.2872$ (4) Å
$V = 1199.32$ (6) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.309$ Mg m^{-3}
Melting point: 434 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1560 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Prism, colourless
0.50 × 0.40 × 0.40 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.953$, $T_{max} = 0.962$
8608 measured reflections
2343 independent reflections
2235 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.9°$
$h = -11 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.068$
$S = 1.04$
2343 reflections
158 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.2043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.045 (6)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.884134.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.16781 (9)	0.17367 (8)	−0.01057 (7)	0.0201 (2)
O2	0.10135 (9)	0.36301 (8)	0.07462 (7)	0.0225 (2)
O3	0.03946 (10)	0.44551 (10)	0.23535 (9)	0.0360 (3)
O4	0.46337 (9)	0.08915 (8)	0.27867 (7)	0.0235 (2)
C1	0.21985 (13)	0.29930 (11)	0.02444 (10)	0.0184 (3)
C2	0.12818 (14)	0.39484 (12)	0.18079 (11)	0.0228 (3)
C3	0.27705 (13)	0.36048 (12)	0.20951 (10)	0.0216 (3)
H31	0.2807	0.3129	0.2795	0.027*
H32	0.3358	0.4397	0.2155	0.027*
C4	0.32810 (12)	0.27457 (11)	0.11621 (10)	0.0172 (3)
H4	0.4266	0.2959	0.0934	0.021*
C5	0.30737 (12)	0.13117 (11)	0.13465 (10)	0.0162 (2)
C6	0.37348 (12)	0.04866 (12)	0.21468 (10)	0.0167 (3)
C7	0.32064 (13)	−0.09032 (12)	0.21643 (10)	0.0184 (3)
H71	0.2375	−0.0952	0.2649	0.023*
H72	0.3950	−0.1463	0.2480	0.023*
C8	0.27957 (13)	−0.14482 (11)	0.10420 (10)	0.0175 (3)
C9	0.17135 (13)	−0.05357 (11)	0.04923 (10)	0.0180 (3)
H91	0.1627	−0.0758	−0.0289	0.022*
H92	0.0778	−0.0648	0.0836	0.022*
C10	0.21803 (12)	0.08331 (11)	0.06087 (9)	0.0163 (3)
C11	0.21468 (14)	−0.27965 (12)	0.11981 (12)	0.0252 (3)
H111	0.1899	−0.3162	0.0487	0.032*
H112	0.1297	−0.2725	0.1647	0.032*
H113	0.2830	−0.3366	0.1560	0.032*
C12	0.41107 (14)	−0.15744 (12)	0.03283 (11)	0.0255 (3)
H121	0.3847	−0.1938	−0.0380	0.032*
H122	0.4792	−0.2150	0.0684	0.032*
H123	0.4535	−0.0715	0.0224	0.032*
C13	0.26637 (16)	0.37458 (13)	−0.07371 (11)	0.0273 (3)
H131	0.1876	0.3821	−0.1249	0.034*
H132	0.3449	0.3293	−0.1090	0.034*
H133	0.2970	0.4615	−0.0514	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0239 (5)	0.0165 (4)	0.0198 (5)	0.0007 (3)	−0.0071 (4)	0.0029 (3)
O2	0.0182 (4)	0.0236 (4)	0.0258 (5)	0.0048 (4)	−0.0010 (4)	−0.0006 (4)
O3	0.0276 (5)	0.0399 (6)	0.0405 (6)	0.0016 (5)	0.0124 (5)	−0.0100 (5)

O4	0.0222 (5)	0.0247 (5)	0.0235 (5)	−0.0021 (4)	−0.0091 (4)	0.0003 (4)
C1	0.0190 (6)	0.0159 (5)	0.0203 (6)	0.0016 (5)	0.0010 (5)	−0.0002 (5)
C2	0.0232 (7)	0.0187 (6)	0.0264 (7)	−0.0034 (5)	0.0041 (5)	−0.0013 (5)
C3	0.0253 (6)	0.0178 (6)	0.0217 (6)	−0.0012 (5)	−0.0001 (5)	−0.0029 (5)
C4	0.0165 (6)	0.0164 (6)	0.0186 (6)	−0.0005 (4)	0.0003 (5)	0.0018 (5)
C5	0.0151 (6)	0.0167 (5)	0.0167 (5)	0.0007 (4)	0.0008 (5)	−0.0001 (4)
C6	0.0144 (6)	0.0203 (6)	0.0154 (6)	0.0006 (4)	0.0009 (5)	−0.0008 (5)
C7	0.0166 (6)	0.0204 (6)	0.0183 (6)	−0.0008 (5)	−0.0040 (5)	0.0044 (5)
C8	0.0171 (6)	0.0159 (5)	0.0195 (6)	0.0003 (5)	−0.0037 (5)	0.0015 (5)
C9	0.0191 (6)	0.0180 (6)	0.0169 (6)	0.0001 (5)	−0.0037 (5)	−0.0005 (5)
C10	0.0144 (5)	0.0186 (6)	0.0158 (6)	0.0030 (5)	−0.0006 (5)	0.0026 (4)
C11	0.0247 (6)	0.0182 (6)	0.0327 (7)	−0.0016 (5)	−0.0080 (6)	0.0033 (5)
C12	0.0237 (7)	0.0222 (6)	0.0306 (7)	0.0038 (5)	0.0035 (5)	−0.0025 (5)
C13	0.0358 (7)	0.0242 (7)	0.0220 (6)	−0.0015 (5)	0.0007 (6)	0.0057 (5)

Geometric parameters (Å, º)

O1—C10	1.3646 (14)	C7—H71	0.99
O1—C1	1.4492 (14)	C7—H72	0.99
O2—C2	1.3689 (16)	C8—C12	1.5303 (17)
O2—C1	1.4399 (15)	C8—C11	1.5300 (16)
O3—C2	1.1956 (16)	C8—C9	1.5464 (16)
O4—C6	1.2324 (15)	C9—C10	1.4835 (17)
C1—C13	1.4997 (17)	C9—H91	0.99
C1—C4	1.5461 (17)	C9—H92	0.99
C2—C3	1.4978 (18)	C11—H111	0.98
C3—C4	1.5265 (16)	C11—H112	0.98
C3—H31	0.99	C11—H113	0.98
C3—H32	0.99	C12—H121	0.98
C4—C5	1.5059 (16)	C12—H122	0.98
C4—H4	1.00	C12—H123	0.98
C5—C10	1.3351 (17)	C13—H131	0.98
C5—C6	1.4426 (16)	C13—H132	0.98
C6—C7	1.5156 (16)	C13—H133	0.98
C7—C8	1.5388 (17)		

C10—O1—C1	107.34 (9)	H71—C7—H72	107.6
C2—O2—C1	111.83 (10)	C12—C8—C11	108.83 (10)
O2—C1—O1	105.50 (9)	C12—C8—C7	109.77 (10)
O2—C1—C13	109.81 (10)	C11—C8—C7	108.66 (10)
O1—C1—C13	108.80 (10)	C12—C8—C9	110.02 (10)
O2—C1—C4	106.33 (9)	C11—C8—C9	109.78 (10)
O1—C1—C4	107.21 (9)	C7—C8—C9	109.76 (9)
C13—C1—C4	118.43 (11)	C10—C9—C8	109.65 (10)
O3—C2—O2	120.52 (12)	C10—C9—H91	109.7
O3—C2—C3	129.37 (12)	C8—C9—H91	109.7
O2—C2—C3	110.08 (11)	C10—C9—H92	109.7
C2—C3—C4	105.00 (10)	C8—C9—H92	109.7

C2—C3—H31	110.7	H91—C9—H92	108.2
C4—C3—H31	110.7	C5—C10—O1	114.02 (10)
C2—C3—H32	110.7	C5—C10—C9	127.24 (11)
C4—C3—H32	110.7	O1—C10—C9	118.74 (10)
H31—C3—H32	108.8	C8—C11—H111	109.5
C5—C4—C3	114.40 (10)	C8—C11—H112	109.5
C5—C4—C1	100.62 (9)	H111—C11—H112	109.5
C3—C4—C1	103.99 (9)	C8—C11—H113	109.5
C5—C4—H4	112.3	H111—C11—H113	109.5
C3—C4—H4	112.3	H112—C11—H113	109.5
C1—C4—H4	112.3	C8—C12—H121	109.5
C10—C5—C6	121.44 (11)	C8—C12—H122	109.5
C10—C5—C4	110.01 (10)	H121—C12—H122	109.5
C6—C5—C4	128.51 (11)	C8—C12—H123	109.5
O4—C6—C5	122.45 (11)	H121—C12—H123	109.5
O4—C6—C7	122.60 (11)	H122—C12—H123	109.5
C5—C6—C7	114.92 (10)	C1—C13—H131	109.5
C6—C7—C8	114.51 (10)	C1—C13—H132	109.5
C6—C7—H71	108.6	H131—C13—H132	109.5
C8—C7—H71	108.6	C1—C13—H133	109.5
C6—C7—H72	108.6	H131—C13—H133	109.5
C8—C7—H72	108.6	H132—C13—H133	109.5

C2—O2—C1—O1	120.97 (10)	C1—C4—C5—C6	−176.76 (11)
C2—O2—C1—C13	−121.94 (11)	C10—C5—C6—O4	174.96 (11)
C2—O2—C1—C4	7.32 (12)	C4—C5—C6—O4	−2.42 (19)
C10—O1—C1—O2	−104.33 (10)	C10—C5—C6—C7	−6.91 (16)
C10—O1—C1—C13	137.90 (10)	C4—C5—C6—C7	175.71 (11)
C10—O1—C1—C4	8.71 (12)	O4—C6—C7—C8	−147.51 (11)
C1—O2—C2—O3	−178.12 (11)	C5—C6—C7—C8	34.36 (14)
C1—O2—C2—C3	3.55 (14)	C6—C7—C8—C12	66.75 (13)
O3—C2—C3—C4	168.91 (13)	C6—C7—C8—C11	−174.33 (10)
O2—C2—C3—C4	−12.95 (13)	C6—C7—C8—C9	−54.28 (13)
C2—C3—C4—C5	−92.39 (12)	C12—C8—C9—C10	−75.43 (12)
C2—C3—C4—C1	16.37 (12)	C11—C8—C9—C10	164.83 (10)
O2—C1—C4—C5	103.98 (10)	C7—C8—C9—C10	45.45 (13)
O1—C1—C4—C5	−8.49 (11)	C6—C5—C10—O1	−178.35 (10)
C13—C1—C4—C5	−131.95 (11)	C4—C5—C10—O1	−0.53 (14)
O2—C1—C4—C3	−14.69 (11)	C6—C5—C10—C9	1.03 (19)
O1—C1—C4—C3	−127.17 (10)	C4—C5—C10—C9	178.85 (11)
C13—C1—C4—C3	109.37 (12)	C1—O1—C10—C5	−5.33 (13)
C3—C4—C5—C10	116.43 (11)	C1—O1—C10—C9	175.23 (10)
C1—C4—C5—C10	5.63 (13)	C8—C9—C10—C5	−21.53 (17)
C3—C4—C5—C6	−65.96 (16)	C8—C9—C10—O1	157.83 (10)